Appendix G

Tank V-9 Analytical Sample Results Report

Appendix G

Tank V-9 Analytical Results Report

G-1. INTRODUCTION AND BACKGROUND

This report presents the analytical results for samples collected from one of the remediation sites addressed under this document—the *Comprehensive Remedial Design/Remedial Action Work Plan for the Test Area North, Waste Area Group 1, Operable Unit 1-10, Group 2 Sites*—specifically Tank V-9, which is part of Technical Support Facility-18 (TSF-18). TSF-09 (Tanks V-1, V-2, and V-3) and TSF-18 (Tank V-9), the V-Tanks, are situated in an open area east of Test Area North-616 (TAN-616) and north of TAN-607. Waste was transferred from the TAN-616 evaporator pit sump and pump room sump, the TAN-607 laboratory drain, the TAN-607 Warm/Hot Shop drain, and TSF-21 (Valve Pit No. 2) through the TAN-1704 valve pit to Tank V-9. The overflow from Tank V-9 drained to the TSF-09 tanks (Idaho National Engineering and Environmental Laboratory [INEEL] 2001).

Tank V-9 was sampled from April 30 to May 8, 2001. Eight samples and one duplicate—for a total of nine samples—were collected for analysis. The primary objective of this sampling effort was to provide isotopic analyses that would support the Tank V-9 criticality evaluation. In addition to the uranium-234/235/238 analysis, the samples were also analyzed for percent moisture, bulk density, and toxicity characteristic leaching procedure (TCLP) metals. Additional isotopic uranium analyses were performed in August 2001, and the results are included in this report. The following sections provide brief descriptions of Tank v-9 and previous characterization efforts.

G-1.1 TSF-18, Contaminated Tank southeast of Tank V-3

TSF-18 includes a single conical-shaped sump tank (Tank V-9), tank contents, an aboveground sand filter, ancillary piping immediately in the vicinity of the tank, and surrounding contaminated soil. The abandoned underground storage tank is located in the open area between the TAN-616 and TAN-633 buildings and is adjacent to the southeast corner of TSF-09.

Tank V-9 was installed in 1953 as part of the V-Tank radioactive waste collection system. The 1,514-L (400-gal) stainless steel sump tank is approximately 1.06 m (42 in.) in diameter in the center and extends approximately 2.1 m (7 ft) to the tip of the cone. The top of Tank V-9 is approximately 2.1 m (7 ft) below ground surface (bgs) and is accessible by a 15.2-cm (6-in.) diameter vent pipe that extends to ground surface. A baffle is located in the tank near the inlet ports. Tank V-9 has two subsurface inlet lines that received wastewater from several TAN sources via the TAN-1704 valve pit. One subsurface outlet line discharged overflow from Tank V-9 to Tanks V-1, V-2, and V-3.

G-1.2 1996 Tank V-9 Characterization Results

The 1996 remedial investigation/feasibility study estimated that approximately 750 to 950 L (200 to 250 gal) of sludge and 265 L (70 gal) of liquid remain in the conical tank. The volume of material located behind the baffle was not known. The total waste material volume inside the tank was estimated at 1,216 L (320 gal) (Blackmore 1998).

In March 1996, Tank V-9 was sampled and 4 L (1.1 gal) of liquid were collected from a location in the tank 3.05 m (10 ft) bgs and an estimated 1.07 m (3.5 ft) above the conical tank bottom. The sample was collected with a peristaltic pump fitted with Teflon tubing and inserted into the tank through a 15.2-cm (6-in.) pipe extending from the ground surface into the top of the tank at the center. The collected liquid, dark brown in color and containing significant amounts of fine particulate matter, was shipped to an onsite laboratory for analysis. The requested analyses for the sample were anions (Cl, NO_3, NO_2, PO_4, SO_4, F, and Br), total halogens, total organic carbon, total suspended solids, pH, Contract Laboratory Program (CLP) metals (with Sn, B, and Si added to the target analyte list), polychlorinated biphenyls (PCBs), volatile organic compounds, semivolatile organic compounds, gamma spectroscopy, U/Pu isotopes, and other isotopes including Cm, Am, Sr, Np, Ra, and H_3 (Department of Energy Idaho Operations Office [DOE-ID] 1997).

In April and June of 1996, the solids in Tank V-9 were sampled using a long-handled device with a detachable sample bomb. The sampling device was inserted into the tank solids, and the sample was collected by sliding a 10.2-cm (4-in.) length of aluminum housing over the material using a T-handle. Outside the tank, the collected material was transferred from the bomb into a stainless steel pan and then into sample jars (two 250-mL sample containers during the first sampling event) with a stainless steel spoon. The tank solids, which had the consistency of mud and contained a large amount of organic debris such as twigs and straw, were collected from a location 4.1 m (13.5 ft) bgs and an estimated 15.2 cm (6 in.) to 0.3 m (1 ft) above the tank bottom. During the second sampling event performed in the same manner in June of 1996, 1,000 mL of tank solids were collected. The majority of the collected material was submitted to an onsite laboratory for the following analyses (in duplicate): anions (Cl, NO_3, NO_2, PO_4, SO_4, F, and Br), total halogens, total organic carbon, percent moisture, pH, particle-size distribution, density, CLP metals (with Sn, B, and Si added to the target analyte list), volatile organic compounds, gamma spectroscopy, U/Pu isotopes, and other isotopes including Cm, Am, Sr, Np, and Ra. Additional sample material was stored under refrigeration until it was shipped to a commercial laboratory in October 1996, for analysis of the CLP target compound list that included semivolatile organic compounds and PCBs using SW-846 methods (DOE-ID 1997).

The analytical results for the liquid sample and two sludge samples collected from Tank V-9 in 1996 are presented in the remedial investigation/feasibility study and also in Appendix H of this Remedial Design/Remedial Action Work Plan. The results of the sampling and analysis indicate that the contents of Tank V-9 are similar in chemical nature to those of Tanks V-1, V-2, and V-3. The sample results reported high concentrations of organic compounds (e.g., trichloroethene and PCBs) and radionuclides (e.g., Cs, U, Am, Pu, and H_3) (DOE-ID 1997). Because of the high concentration of fissile materials in the tank, a criticality evaluation was conducted in 1998. The evaluation was not conclusive in determining if the mass of fissile material in Tank V-9 was sufficient for a criticality event and recommended that additional sampling be conducted (Blackmore 1998).

Based on the recommendation from the 1998 criticality evaluation, nine samples (including one duplicate) were collected from Tank V-9 in April and May 2001, with four of the samples collected from behind the baffle. The remaining sections of this report detail the following aspects of the 2001 sampling effort: sampling equipment and testing, sampling procedures and details, analytical data summary, and lessons learned. Summaries of the raw analytical data and data validation reports are provided in Attachment G-1.

G-2. SAMPLING EQUIPMENT AND TESTING

The original concept proposed for the Tank V-9 sampling equipment was that of a flexible composite liquid waste sampler (COLIWASA) to be diverted to the backside of the baffle through a cable-articulated pipe section. The first full-scale mock-up investigated the feasibility of double articulation of the sampler guide to prevent the sampling device from scraping the wall of the tank while collecting the samples. Testing indicated that there simply was not enough headspace in the tank for double articulation. Consideration was given to the possibility of collecting a single continuous top-to-bottom sample instead of several discrete samples. This technique would have produced less tank disturbance, but the wide range of possible sludge consistencies might have clogged the sampling tube.

Discrete samples require the use of a valved sampler that could be lowered to a known sampling interval, opened, filled, and remotely closed. A commercially available Discrete Sludge Sampler was purchased and tested in a variety of simulated sludges at the Science Applications International Corporation's STAR Center. The simulated sludge consisted primarily of local clay soil and water in various proportions. The valve mechanism failed to operate properly in many of the thicker test sludges.

Project engineers designed a new sampler with a pointed piston valve mechanism. The new sampler performed well in a wide variety of sludges and liquids. The sampler was made from readily available stainless steel and polyvinyl chloride (PVC) pipe fittings. The body of the sampler was a 7-in. long piece of 2-in. schedule 80 PVC pipe. The pipe was threaded into a modified stainless steel bell reducer, which had a threaded attachment for the handle and a pair of setscrews to lock a ½-in. nylon rod that operated the piston at the other end of the pipe (Figure G-1). The sampler would hold a maximum volume of 320 mL; the target sample volume was 200 to 300 mL. Criticality concerns dictated that no more than 5.5 L of material could be removed from the tank during the entire sampling effort. The sampler was attached to a 7-ft flexible hollow handle containing a flexible rod for valve actuation. These, in turn, were connected to shorter sections of a rigid handle and rod to navigate the small-diameter pipe from the tank, up to ground level, and into the glove bag at the samplers' station. An aluminum guide track with a cable-actuated hinge was used to direct the sampler either straight down for the on-axis samples or offset for the off-axis samples. A lamp and tiny video camera were lowered into the tank for initial reconnaissance and continuous monitoring of the sampling activities.

All equipment was built full scale for rigorous testing and operator training at the STAR Center. A platform was built to simulate ground level with the tank mock-up in full view below. During the testing and training phases, minor modifications and fine-tuning improved the equipment. Before mobilization of the equipment to the V-Tank site was authorized, several demonstrations of the equipment were held; the sampling operators were trained in every aspect of setup, operation, and disposal of the sampling equipment, with special emphasis on full containment of the samples and equipment within the glove bag enclosure. Once the final version of the detailed procedure was completed, more training was conducted, video-taped, and timed to document the ability to comply verbatim with each step of the procedure, and to estimate the stay-times that would be required in the radiation field. As low as reasonably achievable (ALARA) practices were emphasized throughout the training.

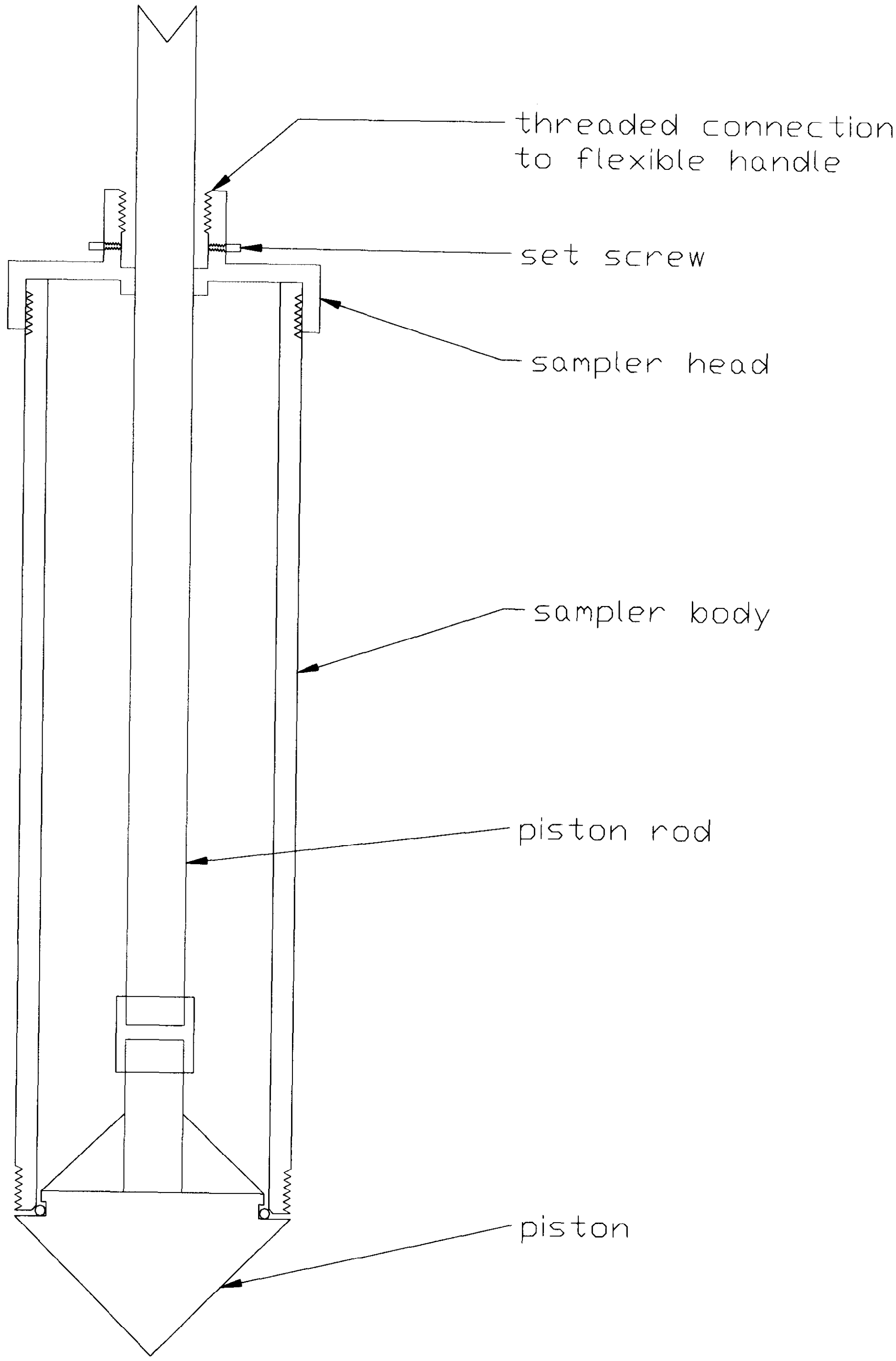

Figure G-1. Sampler Assembly Detail.

G-3. TANK V-9 SAMPLING PROCEDURES AND DETAILS

G-3.1 Sampling Procedures

The sampling procedures specified in the technical procedure "Tank V-9 Sampling Procedure," (WESTON 2001) were generally segregated into three sections: setup, sampling, and disassembly. Consensus was that the setup and disassembly portions were to be "general intent" procedures that could be less rigorously defined with allowances for operator discretion and leeway in the order of operations. The sampling steps that include any steps that disturbed the tank contents in any way required an extremely detailed description with verbatim compliance and a check-off spot for completion of each step. Because of a possible criticality, no chances could be taken that an inadvertently skipped step in the procedure could cause a problem. The procedure is briefly summarized as follows:

1. Build a tent over the tank access
2. Add on extension pipes from ground level up to a comfortable working height
3. Insert lamp and camera for initial reconnaissance and field-verification of dimensions
4. Insert the aluminum guide channel
5. Assemble the glove bag
6. Collect and recover the samples, passing them out of the glove bag into approved shipping containers
7. Decontaminate the equipment
8. Pass equipment out of the glove bag into waste storage containers
9. Disassemble the glove bag
10. Remove extension pipes
11. Remove tent.

Execution followed the written procedure with very few field modifications:

1. The sampling tent was installed without incident. The 10 by 12-ft tent was built on a wooden platform; its primary function was to protect the samplers and equipment from wind and rain.
2. Samplers installed extension pipes. These pipes raised the tank access from below grade level to waist height and had connections for a high-efficiency particulate air vacuum filter and continuous gas monitoring of the tank.
3. The lamp and camera were installed to verify that initial conditions matched those photographed in 1996. The camera-mounting rod was used to measure the actual depth from the top of the extension pipes to the top of the tank for comparison with the assumed depth. Based upon existing information, the assumed depth to the top of the tank had been estimated to be 10 ft, 9.25 in., and the actual depth was measured at 10 ft, 11.25 in. A hold point in the procedure allowed for overnight re-manufacture of some sampling equipment to match the measured depth.

4. The aluminum guide channel was inserted, and its actuator tested. Initial testing of the articulated section disturbed the surface of the water in the tank, leading to the determination that the level in the tank was at least an inch higher than previously seen in 1996.

5. The glove bag was reassembled in the same orientation with respect to the baffle, as it was for all of the training exercises. The soft-sided glove bag was suspended from a steel and wood frame. Overall dimensions were 24 in. wide, 48 in. long, and 30 in. high (Figure G-2). Directly above the tank access was an 8-ft tall sealed "chimney" that provided containment for the long flexible handle. During sample recovery, the handle could be stored above the "trap door" in the chimney, leaving only the sampler body in the glove bag (Figure G-3). The floor of the glove bag supported a vise that was used to clamp the sampler/handle at any elevation.

6. The samples were collected following verbatim compliance with the steps laid out in the work order package. The general procedure was to lower the sampler into the tank headspace by adding 24-in. handle segments, as needed. The guide channel was then articulated to divert the sampler to the backside of the tank baffle. The sampler was then lowered to the beginning of the sample interval. At this point the piston was extended, opening the sampler. The material was allowed to slump back into the void created by the piston. Finally, the sampler body was advanced to "catch up" to the piston, capturing the sample inside the PVC pipe section. Once the sampler was raised up to the glove bag, threaded PVC caps replaced the pointed piston and the handle attachment. The sample was inspected briefly to confirm that sufficient material had been collected. In one instance (1RD005), the sampler had not been properly sealed and there was virtually no sample. The sampling procedure allowed the operators to make another collection attempt from the same location. In three instances, field changes to the sampling procedure were needed that required multiple INEEL approvals.

   The first field change addressed the difficulty encountered in pushing the sampler to Zone 5 (for Sample 1RD006). In the first attempt to collect this sample, the operators encountered layers of crusty sediment behind the baffle. Because this crusty material was substantially different from the practice sludge, the operators decided not to risk damage to the sampling equipment. The sampling procedure was revised to allow them to abandon this sample, collect the remaining samples, and return to Zone 5 at a later date for another attempt to collect 1RD006. After encountering the same crusty layers at the same depths in collecting the two samples from Zone 4, the operators agreed that they could have collected Sample 1RD006 without damaging the sampler.

   The second change was incorporated to give the sampling operators some discretion in further attempts to collect Sample 1RD006. The change authorized a second attempt to collect the sample from the exact same location as the first attempt. In the event of another failure, this change allowed third and fourth attempts to collect a sample from slightly different locations within the boundaries of Zone 5. The second attempt in the original location was successful in collecting the sample, and the remaining options were not exercised.

   The third change allowed a minor deviation in the recovery of the last sample (1RD006). As the sampler was being withdrawn from the tank, it slipped out of the aluminum guide track and became stuck in the extension pipe. Verbatim compliance prohibited re-alignment, as it would require lowering the sample back into the headspace of the tank. The change recognized that this action would not further disturb the tank contents or compromise the sample itself. The sampler was realigned, and the sample recovered without further incident.

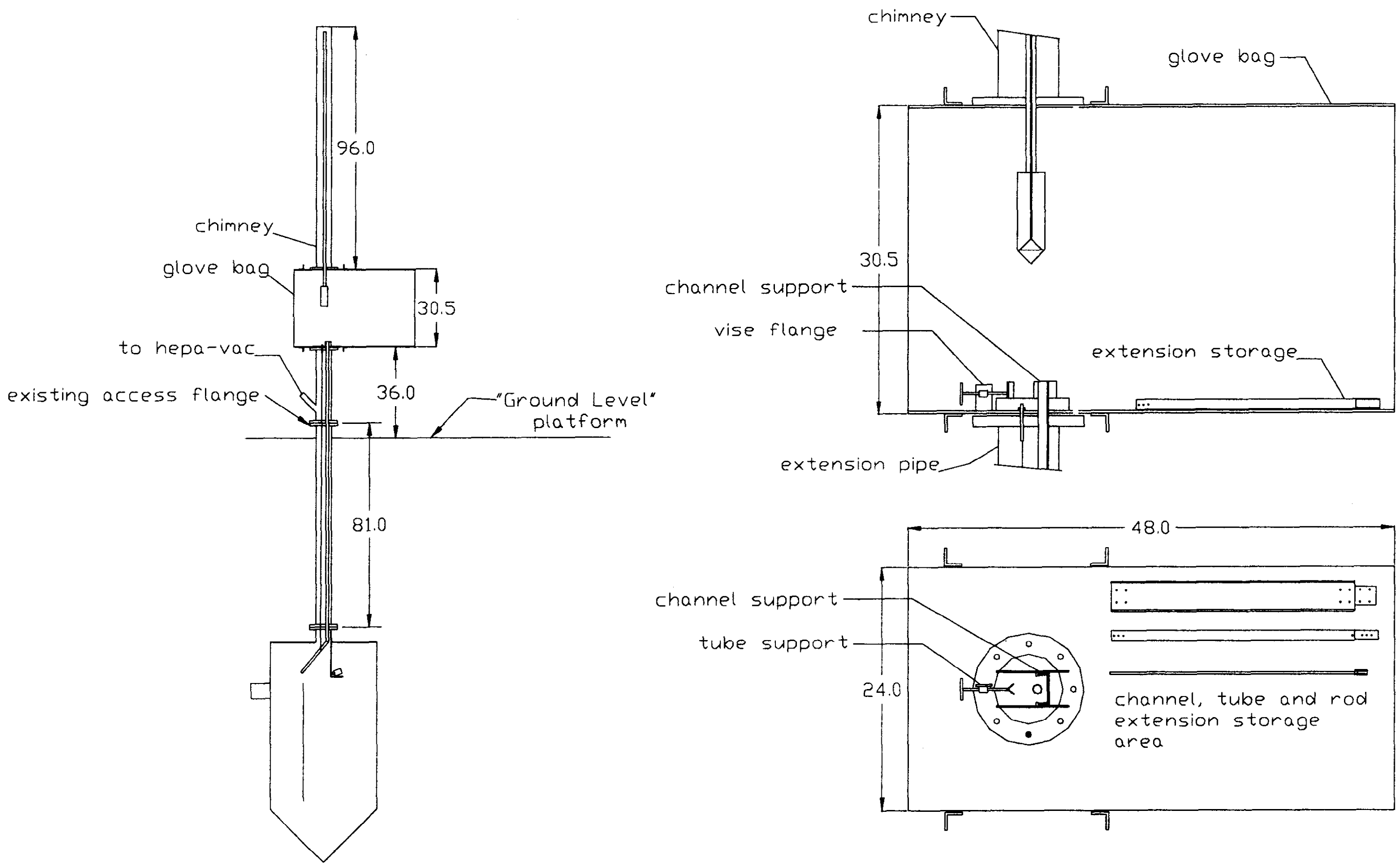

Figure G-2. Sample System and Glove Bag Detail.

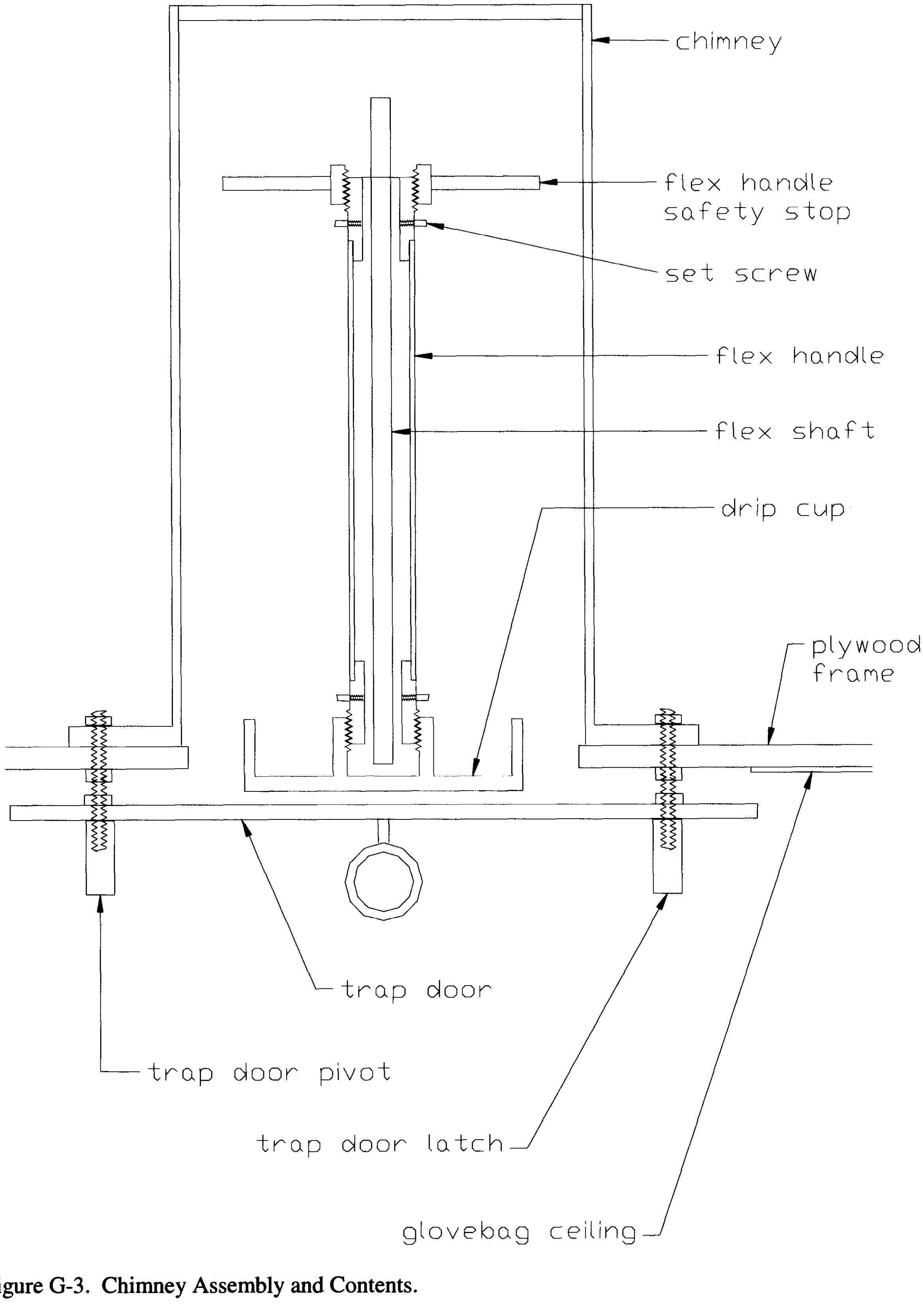

Figure G-3. Chimney Assembly and Contents.

In all cases, the samples were recovered and transferred out of the glove bag. At each recovery, initial measurements were taken to confirm that the radiological limits specified in the radiological work permit were not exceeded. The glove bag was cleaned up and waste material was transferred out before each subsequent sample attempt was initiated.

7. When all samples had been recovered, the equipment was cleaned of gross contamination. The equipment was to be saved for further sampling, if necessary.

8. Most of the equipment had been designed to allow for disassembly within the glove bag without breaking containment. The long, flexible handle was cut into 2-ft lengths. Everything was removed from the glove bag except the two flanges that were too large to fit through the pass-out sleeve.

9. The glove bag was then collapsed around these flanges and packaged in a drum for disposal.

10. The extension pipes were removed and placed in a drum for disposal. One 12-in. section of pipe was left in place to raise the tank access above ground level and was capped with a blind flange.

11. The tent was removed.

G-3.2 Sample Details

Beginning on April 30, 2001, nine samples (including a quality control duplicate) were collected from eight sample zones (Figure G-4) within Tank V-9. The quality control duplicate was taken from Zone 4. The six off-axis zones are each approximately 18 in. long, while the two on-axis sample zones are each approximately 27 in. long. Table G-1 specifies the sample number, location, depth, and date of collection. Individual sample volumes were intended to be between 200 and 300 mL, with a maximum total sample volume of 2.9 L, well within the specified limit of 5.5 L. The sampling procedure included a check to ensure that the sample container was at least half full (160 mL). Weights or volumes of the samples were not recorded due to the ALARA consideration. Therefore, the sample size is reported as approximately 200 to 300 mL. The sample depths in Table G-1 are measured from the top of the tank to the top of the sampler tube.

Table G-1. Sample Numbers, Locations, Depths, and Collection Dates.

Sample Number	Sample Location	Depth from Top of Tank (in.)	Sample Collection Date
1RD001	Zone 1; on tank center line axis	20	April 30, 2001
1RD002	Zone 2; on tank center line axis	56	April 30, 2001
1RD003	Zone 3; off-axis, behind baffle	20	May 1, 2001
1RD004	Zone 4; off-axis, behind baffle	38	May 2, 2001
1RD005	Zone 4; off-axis, behind baffle (duplicate)	38	May 3, 2001
1RD006	Zone 5; off-axis, behind baffle	56	May 8, 2001
1RD007	Zone 6; off-axis, opposite baffle	20	May 3, 2001
1RD008	Zone 7; off-axis, opposite baffle	38	May 3, 2001
1RD009	Zone 8; off-axis, opposite baffle	56	May 7, 2001

Zones 1 and 2 were shallow and deep, respectively, on the centerline of the tank. These samples were collected first such that materials from the other zones would not drip on the surface of Zone 1 and cross-contaminate the samples. Originally, the procedure called for samples to be taken from Zones 3, 4, and 5, in that order, all from the same planimetric position. A duplicate of the Zone 4 sample would then be collected approximately 5 in. offset from this position by rotating the guide channel 10 degrees. During full-scale testing at the STAR Center, it was noted that the duplicate always had a higher sludge-to-water ratio than the original. This was attributed to the action of removing the Zone 3 sample, which created a water channel down toward the original Zone 4 sample location. The final procedure specified that Zone 5 would be sampled after Zone 3 and also allowed the Zone 4 samples to be collected under identical conditions, each 10 degrees (left and right) from the Zone 3 and 5 sampling locations. The last samples to be collected would then be from Zones 6, 7, and 8. Specific details of the samples are summarized below.

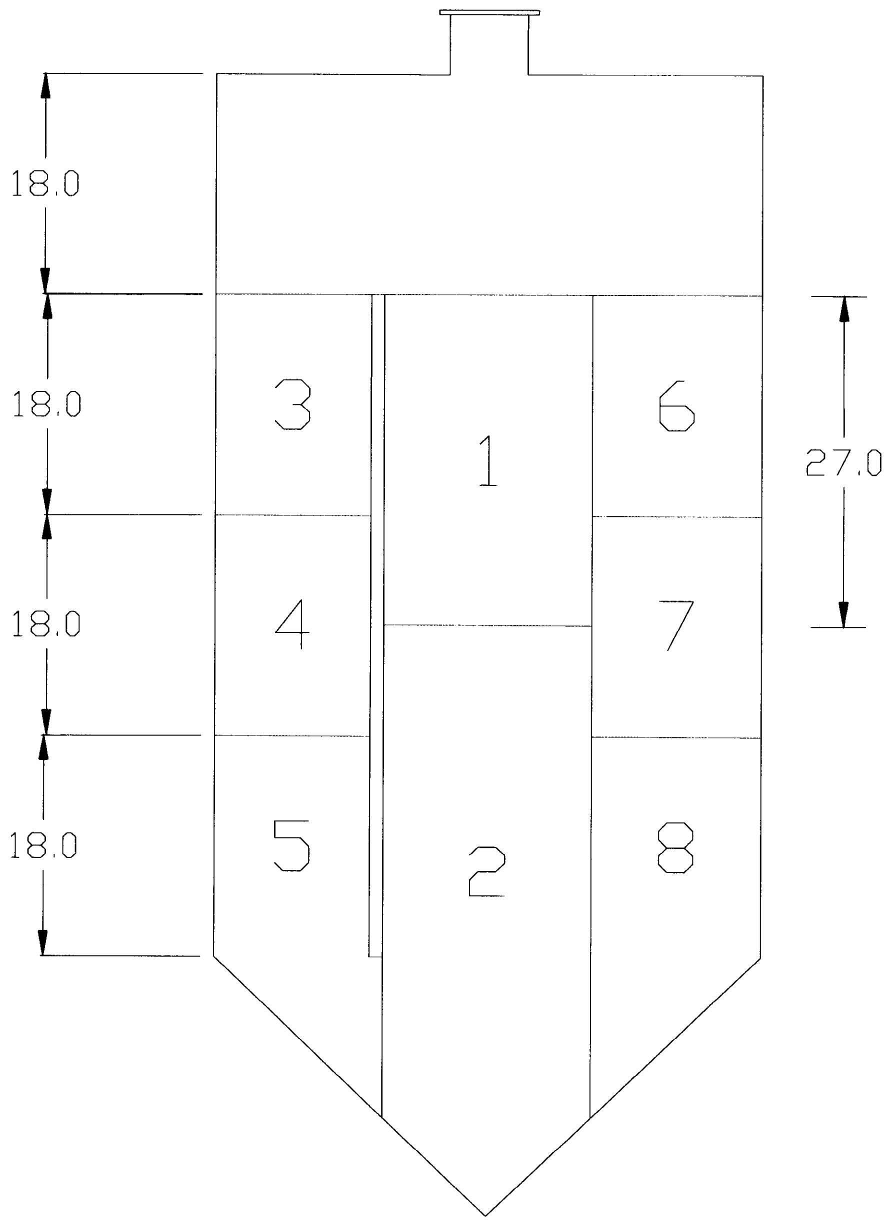

Figure G-4. Tank V-9 Sample Zones.

1. Zone 1 – Sample 1RD001: This sample was collected on the afternoon of April 30, 2001, at 2:00 p.m. The sampler was estimated to be about three-quarters full, and the sample was primarily water. Near contact beta/gamma reading was 3 mR/h.

2. Zone 2 – Sample 1RD002: This sample was also collected on April 30, 2001, at approximately 3:30 p.m. The sampler was inserted to full depth without effort. The sampler was completely full, and the sample had the look and consistency of grease. Near contact beta/gamma reading was 50 mR/h.

3. Zone 3 – Sample 1RD003: This sample was collected on the morning of May 1, 2001, at 11:00 a.m. The sampler was reported to be about one-half full, and the sample was primarily water. Near contact beta/gamma reading was 10 mR/h.

4. Zone 5 – Sample 1RD006: This sample was first attempted on the morning of May 1, 2001. The operators reported significant resistance while attempting to reach Zone 5. Several distinct crusty layers were encountered in Zones 4 and 5. Because the sampling team did not want to risk damaging the sampling apparatus at this early stage of the sampling, the Zone 5 sample was abandoned. The procedure was changed to allow the sampling team to collect the remaining samples before returning to Zone 5.

5. Zone 4 – Sample 1RD004: This sample was collected on the afternoon of May 2, 2001, at about 2:00 p.m. The sampler was full to within 1 in. of the top. The sample, which looked like a purple dye, contained sandy material in the threads of the sample body. Near contact beta/gamma reading was 103 mR/h. The operators reported the same layered crusty conditions noted for the attempt in Zone 5.

6. Zone 4 – Sample 1RD005: This sample was first attempted on the afternoon of May 2, 2001. The sample was captured inside the sampler body, but was lost when the piston lost its seal. During retrieval, the wrong setscrew was inadvertently loosened. The procedure steps allowing a second attempt for "insufficient sample" were followed. The second attempt at about 11:00 a.m. on May 3, 2001, was successful. The sampler was full to within 3/8 in. of the top, and the sample consisted of a mixture of sludge and a gray liquid. Near contact beta/gamma reading was 80 mR/h. The operators noted the same crusty layers as found in the first attempt at Zone 4.

7. Zone 6 – Sample 1RD007: This sample was collected at about 12:00 p.m. on May 3, 2001. The sampler was full to within 3/8 in. of the top with liquid and sludge. The sample appeared to have some grass mixed in with it. Near contact beta/gamma reading was 9 mR/h.

8. Zone 7 – Sample 1RD008: This sample, collected at about 4:00 p.m. on the afternoon of May 3, 2001, was filled to within ½ in. of the top. There was no appearance of crusty layers in this zone. Near contact beta/gamma reading was 100 mR/h.

9. Zone 8 – Sample 1RD009: This sample, collected May 7, 2001, at around 10:30 a.m., filled the sampler to within 1/8 in. of the top. The sample looked like black grease and contained what appeared to be part of an old rubber glove. Near contact beta/gamma reading was 80 mR/h.

10. Zone 5 – Sample 1RD006 (second attempt): This sample was collected on May 7, 2001, from the same location as the original attempt on May 1. A little extra force was used to push through the last crusty layer. During the retrieval process, the sampler slipped out of the track and wedged in the pipe riser. The procedure was changed to allow for realignment, and the sample was finally recovered on the morning of May 8, 2001, at about 9:00 a.m. The sample looked like grease and included what seemed to be part of a root or stick. Near contact beta/gamma reading was 80 mR/h.

G-4. TANK V-9 ANALYTICAL DATA SUMMARY

The nine samples were delivered to the BWXT Services, Inc., Nuclear Environmental Laboratory Services located in Lynchburg, Virginia. The samples were received at the laboratory from May 2 through May 9, 2001. On May 10, 2001, each sample was placed in a sample tray and homogenized to the extent practical. From each of the nine homogenized samples, five subsamples were collected for isotopic uranium analysis (using alpha spectrometry), as well as percent moisture and bulk density determinations. Additionally, two composites were prepared from the nine homogenized samples by combining approximately 10-mL aliquots from the Zone 1, 3, and 6 samples with 10- to 25-g aliquots from the remaining six samples (BWXT Services 2001a). The composites were analyzed for TCLP metals.

The initial analytical results for the 45 subsamples are presented in Table G-2, and the TCLP metal analytical results for the composites are presented in Table G-3. In addition to the individual data values for the subsamples, Table G-2 also provides maximum, average, standard deviation, and relative percent difference (RPD) values for each group of five subsamples. Specific results are discussed in the following sections. Summaries of the raw data and data validation reports are included in Attachment G-1. Radioanalytical and TCLP metal data validations were performed by Portage Environmental, Inc., in Butte, Montana, and bulk density and moisture content data validations were performed by the INEEL Sample Management Office.

In August 2001, the BWXT Services, Inc., laboratory was requested to perform additional isotopic uranium analyses on the remaining 45 subsample volumes that had been retained by the laboratory. One of the five subsamples from each sampling zone was randomly selected, and an aliquot was collected and split. One of the splits was prepared using acid dissolution, and the other split was prepared using pyrosulfate fusion. Both splits were then analyzed using alpha spectrometry. A duplicate analysis of the Zone 2 fusion split was also performed. Section G-4.4 and Table G-4 present the additional radioanalytical results that were obtained (BWXT Services 2001b), and summaries of the raw data and data validation reports are included in Attachment G-1. The analytical method data validation was performed by Environmental Data Services, Inc., which is located in Indianola, Pennsylvania.

G-4.1 Bulk Density and Moisture Content Results

The bulk density and moisture content analyses were conducted using American Society for Testing and Materials Standards D5057-90 and E949-88, respectively, and the results are presented in Table G-2. For these analyses, the only applicable quality control parameter is the sample duplicate. Since the laboratory essentially performed duplicates (i.e., the five subsamples collected from each original sample) for every sample, data validation evaluated the sample results based on their subsample's RPDs to each other. For each sample, the maximum and minimum values reported for the five subsamples were used for this calculation as follows:

$$RPD = \frac{Max - Min}{Max + Min} \times 200$$

If the RPD was greater than 35% but less than or equal to 50%, the associated sample results were qualified with a "J" validation flag. If the RPD was greater than 50%, the associated sample results were qualified with an "R" validation flag.

Table G-2. Tank V-9 Physical and Radiochemical Analytical Results

Sample Zone	Sample	Subsample Number	Moisture Content (%)	Bulk Density (g/mL)	DVF	U-234 Results (pCi/g)	U-234 Uncertainty (pCi/g)	U-235 Results (pCi/g)	DVF	U-235 Uncertainty (pCi/g)	U-238 Results (pCi/g)	DVF	U-238 Uncertainty (pCi/g)
1	1RD001	1RD001013A	98.0	1.0	—	1.90E+02	1.58E+01	9.94E+00	J[2]	1.36E+00	1.53E+00	—	4.62E-01
1	1RD001	1RD001023A	98.3	1.0	—	1.73E+02	1.44E+01	8.98E+00	J[2]	1.25E+00	1.39E+00	—	4.29E-01
1	1RD001	1RD001033A	97.9	1.0	—	1.91E+02	1.60E+01	1.01E+01	J[2]	1.42E+00	1.69E+00	—	5.11E-01
1	1RD001	1RD001043A	98.0	1.0	—	1.69E+02	1.41E+01	9.49E+00	J[2]	1.31E+00	7.14E-01	J[3]	3.15E-01
1	1RD001	1RD001053A	98.1	1.0	—	1.65E+02	1.36E+01	9.94E+00	J[2]	1.25E+00	1.28E+00	—	3.73E-01
		Maximum	98.3	1.0	—	1.91E+02	1.60E+01	1.01E+01	—	1.42E+00	1.69E+00	—	5.11E-01
		Average	98.1	1.0	—	1.78E+02	1.48E+01	9.69E+00	—	1.32E+00	1.32E+00	—	4.18E-01
		Std. Dev.	0.2	0.0	—	1.21E+01	1.06E+00	4.57E-01	—	7.33E-02	3.72E-01	—	7.64E-02
		RPD (%)	0.4	0.0	—	—	—	—	—	—	—	—	—
2	1RD002	1RD002013A	46.9	0.8	R	1.55E+04	1.25E+03	6.05E+02	J[2]	6.60E+01	1.33E+02	—	2.39E+01
2	1RD002	1RD002023A	46.7	2.7	R	5.33E+03	4.46E+02	2.31E+02	J[2]	3.45E+01	1.14E+02	—	2.26E+01
2	1RD002	1RD002033A	46.1	1.5	R	6.93E+03	5.70E+02	2.55E+02	J[2]	3.49E+01	1.11E+02	—	2.08E+01
2	1RD002	1RD002043A	46.3	1.4	R	1.12E+04	9.08E+02	3.34E+02	J[2]	4.26E+01	1.36E+02	—	2.40E+01
2	1RD002	1RD002053A	47.6	3.4	R	8.85E+03	7.30E+02	3.85E+02	J[2]	4.78E+01	1.00E+02	—	2.05E+01
		Maximum	47.6	3.4	—	1.55E+04	1.25E+03	6.05E+02	—	6.60E+01	1.36E+02	—	2.40E+01
		Average	46.7	2.0	—	9.56E+03	7.81E+02	3.62E+02	—	4.52E+01	1.19E+02	—	2.24E+01
		Std. Dev.	0.6	1.1	—	3.98E+03	3.14E+02	1.49E+02	—	1.29E+01	1.53E+01	—	1.66E+00
		RPD (%)	3.2	123.8	—	—	—	—	—	—	—	—	—
3	1RD003	1RD003013A	97.4	1.0	—	8.42E+01	6.94E+00	4.49E+00	J[2]	5.52E-01	6.69E-01	—	1.81E-01
3	1RD003	1RD003023A	97.1	1.0	—	8.96E+01	7.34E+00	5.22E+00	J[2]	6.09E-01	8.31E-01	—	1.93E-01
3	1RD003	1RD003033A	93.2	1.0	—	8.57E+01	7.02E+00	5.66E+00	J[2]	6.33E-01	8.83E-01	—	1.92E-01
3	1RD003	1RD003043A	100.4	1.0	—	8.62E+01	7.04E+00	4.83E+00	J[2]	5.71E-01	5.91E-01	—	1.58E-01
3	1RD003	1RD003053A	97.0	1.0	—	8.26E+01	6.80E+00	4.21E+00	J[2]	5.14E-01	6.24E-01	—	1.60E-01
		Maximum	100.4	1.0	—	8.96E+01	7.34E+00	5.66E+00	—	6.33E-01	8.83E-01	—	1.93E-01
		Average	97.0	1.0	—	8.57E+01	7.03E+00	4.88E+00	—	5.76E-01	7.20E-01	—	1.77E-01
		Std. Dev.	2.6	0.0	—	2.61E+00	1.98E-01	5.76E-01	—	4.69E-02	1.30E-01	—	1.69E-02
		RPD (%)	7.4	0.0	—	—	—	—	—	—	—	—	—
4	1RD004	1RD004013A	52.5	1.7	J[1]	1.38E+04	1.12E+03	7.19E+02	—	7.42E+01	3.66E+02	—	4.47E+01
4	1RD004	1RD004023A	53.1	1.4	J[1]	2.22E+04	1.78E+03	8.98E+02	—	8.91E+01	3.08E+02	—	3.98E+01
4	1RD004	1RD004033A	43.7	1.6	J[1]	7.44E+03	6.22E+02	3.54E+02	—	4.78E+01	3.03E+02	—	4.31E+01

Table G-2. (continued).

Sample Zone	Sample	Subsample Number	Moisture Content (%)	Bulk Density (g/mL)	DVF	U-234 Results (pCi/g)	U-234 Uncertainty (pCi/g)	U-235 Results (pCi/g)	DVF	U-235 Uncertainty (pCi/g)	U-238 Results (pCi/g)	DVF	U-238 Uncertainty (pCi/g)
4	1RD004	1RD004043A	54.8	1.8	J[1]	1.86E+04	1.51E+03	7.57E+02	—	8.29E+01	3.78E+02	—	5.04E+01
4	1RD004	1RD004053A	39.5	2.2	J[1]	8.41E+03	6.92E+02	3.74E+02	—	4.75E+01	2.38E+02	—	3.51E+01
		Maximum	54.8	2.2	—	2.22E+04	1.78E+03	8.98E+02	—	8.91E+01	3.78E+02	—	5.04E+01
		Average	48.7	1.7	—	1.41E+04	1.14E+03	6.20E+02	—	6.83E+01	3.19E+02	—	4.26E+01
		Std. Dev.	6.7	0.3	—	6.38E+03	5.04E+02	2.43E+02	—	1.96E+01	5.62E+01	—	5.69E+00
		RPD (%)	32.4	44.4	—	—	—	—	—	—	—	—	—
4	1RD005	1RD005013A	55.7	2.4	R	7.03E+03	5.82E+02	4.00E+02	—	4.96E+01	4.14E+02	—	5.08E+01
4	1RD005	1RD005023A	58.5	1.3	R	2.16E+04	1.74E+03	1.22E+03	—	1.16E+02	1.05E+03	—	1.03E+02
4	1RD005	1RD005033A	64.7	1.5	R	8.21E+03	6.74E+02	4.78E+02	—	5.30E+01	4.28E+02	—	4.88E+01
4	1RD005	1RD005043A	55.9	1.5	R	1.32E+04	1.06E+03	7.73E+02	—	7.77E+01	5.61E+02	—	6.03E+01
4	1RD005	1RD005053A	55.2	1.9	R	2.01E+04	1.61E+03	1.14E+03	—	1.08E+02	1.33E+03	—	1.23E+02
		Maximum	64.7	2.4	—	2.16E+04	1.74E+03	1.22E+03	—	1.16E+02	1.33E+03	—	1.23E+02
		Average	58.0	1.7	—	1.40E+04	1.13E+03	8.02E+02	—	8.09E+01	7.57E+02	—	7.72E+01
		Std. Dev.	4.0	0.4	—	6.67E+03	5.28E+02	3.73E+02	—	3.06E+01	4.12E+02	—	3.37E+01
		RPD (%)	15.8	59.5	—	—	—	—	—	—	—	—	—
5	1RD006	1RD006013A	43.6	1.5	—	2.38E+04	1.99E+03	1.13E+03	—	1.34E+02	8.18E+02	—	1.06E+02
5	1RD006	1RD006023A	39.6	1.6	—	2.75E+04	2.30E+03	1.25E+03	—	1.46E+02	9.72E+02	—	1.22E+02
5	1RD006	1RD006033A	37.0	1.8	—	1.73E+04	1.46E+03	1.11E+03	—	1.31E+02	1.19E+03	—	1.37E+02
5	1RD006	1RD006043A	40.8	1.6	—	1.46E+04	1.23E+03	7.47E+02	—	9.57E+01	9.30E+02	—	1.12E+02
5	1RD006	1RD006053A	41.0	1.9	—	1.37E+04	1.17E+03	8.21E+02	—	1.06E+02	7.41E+02	—	9.87E+01
		Maximum	43.6	1.9	—	2.75E+04	2.30E+03	1.25E+03	—	1.46E+02	1.19E+03	—	1.37E+02
		Average	40.4	1.7	—	1.94E+04	1.63E+03	1.01E+03	—	1.23E+02	9.30E+02	—	1.15E+02
		Std. Dev.	2.4	0.2	—	6.02E+03	4.95E+02	2.16E+02	—	2.09E+01	1.71E+02	—	1.49E+01
		RPD (%)	16.4	23.5	—	—	—	—	—	—	—	—	—
6	1RD007	1RD007013A	94.0	1.0	—	5.04E+02	4.14E+01	2.65E+01	—	3.04E+00	1.07E+01	—	1.63E+00
6	1RD007	1RD007023A	94.3	1.1	—	4.80E+02	3.91E+01	2.83E+01	—	3.06E+00	1.01E+01	—	1.48E+00
6	1RD007	1RD007033A	93.9	1.0	—	3.86E+02	3.16E+01	1.88E+01	—	2.31E+00	5.59E+00	—	1.07E+00
6	1RD007	1RD007043A	94.4	1.1	—	5.08E+02	4.16E+01	2.79E+01	—	3.16E+00	1.03E+01	—	1.60E+00
6	1RD007	1RD007053A	94.3	1.0	—	4.48E+02	3.68E+01	2.39E+01	—	2.84E+00	7.82E+00	—	1.37E+00
		Maximum	94.4	1.1	—	5.08E+02	4.16E+01	2.83E+01	—	3.16E+00	1.07E+01	—	1.63E+00
		Average	94.2	1.0	—	4.65E+02	3.81E+01	2.51E+01	—	2.88E+00	8.90E+00	—	1.43E+00
		Std. Dev.	0.2	0.1	—	5.03E+01	4.13E+00	3.91E+00	—	3.40E-01	2.17E+00	—	2.26E-01

Table G-2. (continued).

Sample Zone	Sample	Subsample Number	Moisture Content (%)	Bulk Density (g/mL)	DVF	U-234 Results (pCi/g)	U-234 Uncertainty (pCi/g)	U-235 Results (pCi/g)	DVF	U-235 Uncertainty (pCi/g)	U-238 Results (pCi/g)	DVF	U-238 Uncertainty (pCi/g)
		RPD (%)	0.5	9.5	—	—	—	—	—	—	—	—	—
7	1RD008	1RD008013A	66.9	1.4	—	4.11E+03	3.35E+02	1.73E+02	—	2.16E+01	2.50E+01	—	6.76E+00
7	1RD008	1RD008023A	67.5	1.3	—	4.06E+03	3.30E+02	1.69E+02	—	2.07E+01	1.85E+01	—	5.58E+00
7	1RD008	1RD008033A	66.4	1.3	—	4.45E+03	3.67E+02	1.41E+02	—	1.96E+01	1.64E+01	J^3	5.84E+00
7	1RD008	1RD008043A	66.5	1.2	—	3.72E+03	3.06E+02	1.65E+02	—	2.16E+01	1.87E+01	J^3	6.55E+00
7	1RD008	1RD008053A	67.1	1.3	—	4.64E+03	3.78E+02	1.90E+02	—	2.30E+01	2.95E+01	—	7.34E+00
		Maximum	67.5	1.4	—	4.64E+03	3.78E+02	1.90E+02	—	2.30E+01	2.95E+01	—	7.34E+00
		Average	66.9	1.3	—	4.20E+03	3.43E+02	1.68E+02	—	2.13E+01	2.16E+01	—	6.41E+00
		Std. Dev.	0.4	0.1	—	3.59E+02	2.92E+01	1.77E+01	—	1.26E+00	5.45E+00	—	7.11E-01
		RPD (%)	1.6	15.4	—	—	—	—	—	—	—	—	—
8	1RD009	1RD009013A	62.0	1.6	R	2.75E+03	2.29E+02	1.07E+02	—	1.57E+01	1.22E+01	J^3	4.77E+00
8	1RD009	1RD009023A	62.4	2.3	R	3.34E+03	2.75E+02	1.34E+02	—	1.84E+01	1.11E+01	J^3	4.60E+00
8	1RD009	1RD009033A	61.6	1.1	R	3.98E+03	3.27E+02	1.39E+02	—	1.83E+01	1.48E+01	J^3	5.08E+00
8	1RD009	1RD009043A	59.9	1.7	R	3.36E+03	2.74E+02	9.89E+01	—	1.37E+01	1.38E+01	—	4.43E+00
8	1RD009	1RD009053A	63.4	1.2	R	3.73E+03	3.04E+02	1.26E+02	—	1.69E+01	1.14E+01	J^3	4.40E+00
		Maximum	63.4	2.3	—	3.98E+03	3.27E+02	1.39E+02	—	1.84E+01	1.48E+01	—	5.08E+00
		Average	61.9	1.6	—	3.43E+03	2.82E+02	1.21E+02	—	1.66E+01	1.27E+01	—	4.66E+00
		Std. Dev.	1.1	0.5	—	5.02E+02	4.01E+01	1.98E+01	—	2.26E+00	1.65E+00	—	2.77E-01
		RPD (%)	5.7	70.6	—	—	—	—	—	—	—	—	—

RPD = relative percent difference using maximum and minimum values among the five subsamples from each zone.

DVF = data validation flag. Data validation flags apply to results in the preceding column. If a data validation flag is not shown, the results in the preceding column are unqualified and can be categorized as definitive data with no associated quality control deficiencies.

R = The accuracy of the data is so questionable that it is recommended that the data not be used. For the three bulk density samples 1RD002, 1RD005, and 1RD009, an "R" flag is assigned because the RPDs between the subsamples (using the highest and lowest subsample values) were greater than 50% (i.e., 123.8%, 59.4%, and 70.6%, respectively).

J = estimated value

J^1 = The data are detectable at the reported values, but the reported values are only estimates due to anomalies in the quality control data. For the bulk density sample 1RD004, the "J^1" flag is assigned because the RPD between the subsamples (using the highest and lowest subsample values) was greater than 35% but less than or equal to 50% (i.e., 44.4%).

J^2 = The analysis was performed and radioactivity was detected. However, the result is questionable due to analytical and/or laboratory quality control anomalies and should therefore be used only as an estimated quantity. For the U-235 analyses for samples 1RD001, 1RD002, and 1RD003, one of the three laboratory control sample recovery results (137.7%) was greater than the prescribed limits of 70–130%, which may indicate high biased sample results.

J^3 = The analysis was performed and radioactivity was detected. However, the result is questionable due to analytical and/or laboratory quality control anomalies and should therefore be used only as an estimated quantity. For the seven U-238 subsample analyses assigned the "J^3" flag, the results are statistically positive, but the activities are between 2 and 3 times the associated uncertainty instead of greater than 3 times the uncertainty for unflagged values.

g/mL = grams per milliliter

pCi/g = picocuries per gram

Table G-3. Tank V-9 TCLP Analytical Results.

Sample Number	Analyte	Analytical Method	Results (µg/L)	Data Validation Flag	MDL (µg/L)	Required Detection Limit (µg/L)	Regulatory Level (µg/L)	Exceeds Regulatory Limit
1RD01001TI	Arsenic	ICP--6010A	37.8	U	37.8	250	5,000	NO
1RD01001TI	Barium	ICP--6010A	921	J[1]	4.44	1,000	100,000	NO
1RD01001TI	Cadmium	ICP--6010A	970	J[2]	4.44	50	1,000	NO
1RD01001TI	Chromium	ICP--6010A	234	–	17.8	250	5,000	NO
1RD01001TI	Lead	ICP--6010A	84.4	U	84.4	250	5,000	NO
1RD01001TI	Mercury	CV--7470A	133	–	4	2	200	NO
1RD01001TI	Selenium	ICP--6010A	48.9	U	48.9	50	1,000	NO
1RD01001TI	Silver	ICP--6010A	15.6	U, UJ	15.6	250	5,000	NO
1RD01002TI	Arsenic	ICP--6010A	46.2	B, U[1]	37.8	250	5,000	NO
1RD01002TI	Barium	ICP--6010A	969	J[1]	4.44	1,000	100,000	NO
1RD01002TI	Cadmium	ICP--6010A	1,000	J[2]	4.44	50	1,000	YES
1RD01002TI	Chromium	ICP--6010A	276	–	17.8	250	5,000	NO
1RD01002TI	Lead	ICP--6010A	89.8	B	84.4	250	5,000	NO
1RD01002TI	Mercury	CV--7470A	226	–	4	2	200	YES
1RD01002TI	Selenium	ICP--6010A	64	B, U[1]	48.9	50	1,000	NO
1RD01002TI	Silver	ICP--6010A	15.6	U, UJ	15.6	250	5,000	NO

Method Legend:

MDL = method detection limit

ICP = inductively coupled plasma atomic emission spectrometry

CV = cold vapor atomic absorption spectrometry

Laboratory Concentration Comment (U and B codes) and Data Validation Flag Legend:

B = Analyte concentration greater than the method detection limit (MDL), but less than 10 times the MDL

J = estimated value

J[1] = All barium results have been qualified with a "J[1]" validation flag to denote that the data are detectable at the reported values but that the reported values are only estimates due to low recoveries for continuing calibration verification (89.4% vs. 90–110% recovery criteria), matrix spike (78.5% vs. 80–120% recovery criteria), and matrix spike duplicate (79.0% vs. 80–120% recovery criteria).

J[2] = All cadmium results have been qualified with a "J[2]" validation flag to denote that the data are detectable at the reported values but that the reported values are only estimates due to low recoveries for matrix spike (77.8% vs. 80–120% recovery criteria), matrix spike duplicate (78.6% vs. 80–120% recovery criteria), and analytical spike (-94.8% vs. 75–125% recovery criteria).

U = analyte concentration less than the MDL

U[1] = The arsenic and selenium results for sample 1RD01002TI have been qualified with a "U[1]" validation flag to denote that the sample results are greater than the method detection limits but less than five times the amount of analyte found in the preparation blanks.

UJ = All silver sample results have been qualified with a "UJ" validation flag to denote that the data are non-detectable at the reported values and that the reported values are only estimates due to low recoveries for matrix spike (66.4% vs. 80–120% recovery criteria), matrix spike duplicate (73.4% vs. 80–120% recovery criteria), and analytical spike (64.0% vs. 75–125% recovery criteria).

µg/L = micrograms per liter

Table G-4. Tank V-9 Additional Isotopic Uranium Analyses.

Sample Zone	Sample Number	Sample Size (grams)	Preparation Method	U-234 Results (pCi/g)	DVF	U-234 Uncert. (pCi/g)	U-235 Results (pCi/g)	DVF	U-235 Uncert. (pCi/g)	U-238 Results (pCi/g)	DVF	U-238 Uncert. (pCi/g)
1	1RD05001R9	1.0714	Dissolution	1.36E+02	J	1.12E+01	6.88E+00	J	8.70E-01	7.52E-01	J	2.53E-01
1	1RD05002R9	1.0573	Fusion	1.31E+02	J	1.06E+01	6.78E+00	J	7.98E-01	9.65E-01	J	2.40E-01
			RPD (%)	3.7	—	—	1.5	—	—	24.8	—	—
2	1RD05101R9	0.0103	Dissolution	1.01E+04	J	9.02E+02	4.09E+02	J	6.54E+01	1.60E+02	J	3.76E+01
2	1RD05102R9	0.0106	Fusion	7.87E+03	J	6.76E+02	3.22E+02	J	5.33E+01	1.48E+02	J	3.37E+01
			RPD (%)	24.8	—	—	23.8	—	—	7.8	—	—
2	1RD05102R9	0.0106	Fusion	7.87E+03	J	6.76E+02	3.22E+02	J	5.33E+01	1.48E+02	J	3.37E+01
2-Dup.	0107074-04D	0.01	Fusion	8.46E+03	J	7.33E+02	2.65E+02	J	4.87E+01	3.44E+02	J	5.71E+01
			RPD (%)	7.2	—	—	19.4	—	—	79.7	—	—
3	1RD05201R9	1.1919	Dissolution	7.08E+01	J	6.06E+00	3.85E+00	J	5.27E-01	5.27E-01	J	1.66E-01
3	1RD05202R9	1.0340	Fusion	7.48E+01	J	6.20E+00	4.95E+00	J	6.41E-01	9.49E-01	J	2.39E-01
			RPD (%)	5.5	—	—	25.0	—	—	57.2	—	—
4	1RD05301R9	0.0110	Dissolution	3.08E+04	J	2.54E+03	1.19E+03	J	1.33E+02	3.74E+02	J	5.94E+01
4	1RD05302R9	0.0111	Fusion	3.01E+04	J	2.57E+03	1.15E+03	J	1.31E+02	3.18E+02	J	5.40E+01
			RPD (%)	2.3	—	—	3.4	—	—	16.2	—	—
4	1RD05401R9	0.0265	Dissolution	2.40E+04	J	1.94E+03	1.30E+03	J	1.43E+02	8.45E+02	J	1.04E+02
4	1RD05402R9	0.0258	Fusion	2.33E+04	J	1.88E+03	1.08E+03	J	1.17E+02	7.95E+02	J	9.36E+01
			RPD (%)	3.0	—	—	18.5	—	—	6.1	—	—
5	1RD05501R9	0.0113	Dissolution	5.29E+04	J	4.34E+03	2.35E+03	J	2.39E+02	9.61E+02	J	1.21E+02
5	1RD05502R9	0.0113	Fusion	5.65E+04	J	4.70E+03	2.52E+03	J	2.73E+02	1.06E+03	J	1.45E+02
			RPD (%)	6.6	—	—	7.0	—	—	9.8	—	—
6	1RD05601R9	1.2042	Dissolution	5.67E+02	J	4.56E+01	2.80E+01	J	2.86E+00	1.31E+01	J	1.62E+00
6	1RD05602R9	1.2973	Fusion	5.61E+02	J	4.49E+01	3.09E+01	J	3.05E+00	1.33E+01	J	1.60E+00
			RPD (%)	1.1	—	—	9.8	—	—	1.5	—	—
7	1RD05701R9	0.0258	Dissolution	4.50E+03	J	3.91E+02	1.84E+02	J	2.89E+01	3.06E+01	J	1.06E+01
7	1RD05702R9	0.0283	Fusion	3.93E+03	J	3.26E+02	1.56E+02	J	2.35E+01	2.75E+01	J	8.87E+00

Table G-4. (continued).

Sample Zone	Sample Number	Sample Size (grams)	Preparation Method	U-234 Results (pCi/g)	DVF	U-234 Uncert. (pCi/g)	U-235 Results (pCi/g)	DVF	U-235 Uncert. (pCi/g)	U-238 Results (pCi/g)	DVF	U-238 Uncert. (pCi/g)
			RPD (%)	13.5	—	—	16.5	—	—	10.7	—	—
8	1RD05801R9	0.0263	Dissolution	3.43E+03	J	2.91E+02	1.11E+02	J	2.03E+01	7.34E+00	U	5.36E+00
8	1RD05802R9	0.0256	Fusion	3.49E+03	J	3.02E+02	1.19E+02	J	2.04E+01	1.05E+01	U	5.79E+00
			RPD (%)	1.7	—	—	7.0	—	—	35.4	—	—

Preparation Method Legend: Dissolution—strong mineral acid dissolution; Fusion—molten salt fusion.

DVF = Data validation flag. Data validation flags apply to results in the preceding column.

J = estimated value. The analysis was performed and radioactivity was detected (i.e., the radioanalytical result is statistically positive at the 95% confidence level and is above the minimum detectable activity). However, the result is questionable due to analytical and/or laboratory quality control anomalies (i.e., uranium isotope laboratory control sample recoveries outside acceptable tolerances) and should, therefore, be used only as an estimated (approximated) quantity.

NOTE: The radionuclide is considered to be present in the sample, but the result may not be an accurate representation of the amount of activity actually present in the sample.

U = undetected. The analysis was performed, but no radioactivity was detected (i.e., the radioanalytical result was not statistically positive at the 95% confidence level and/or the result was below its minimum detectable activity). The "U" qualifier flag is also applicable to any result reported as zero (0) (+/- an associated uncertainty).

NOTE: The radionuclide is not considered to be present in the sample.

RPD = relative percent difference

pCi/g = picocuries/gram

Uncert. = Uncertainty

Dup. = Duplicate

As discussed in Section G-3.2, the samples collected from Zones 1, 3, and 6 (the uppermost zones in the tank) were primarily liquid, and the samples from the remaining zones exhibited more of a sludge/paste consistency. These observations are confirmed by the analytical data where the average bulk density for the upper zones is 1.0 g/mL and average moisture content ranges from 94.4% in Zone 6 to 98.1% in Zone 1. RPDs for bulk density and moisture content vary from 0.0 to 9.5% and 0.4 to 7.4%, respectively.

The bulk density and moisture content results for the remaining sample zones (2, 4, 5, 7, and 8) vary considerably more. Average bulk density ranges from 1.3 to 2.0 g/mL, and average moisture content ranges from 40.4 to 66.9%. RPDs for bulk density and moisture content data range from 15.4 to 123.8% and from 1.6 to 32.4%. Based upon the RPD criteria above, the bulk density results for Samples 1RD002, 1RD005, and 1RD009 are assigned an "R" validation flag, and the bulk density for 1RD004 is assigned a "J" validation flag.

G-4.2 Isotopic Uranium Activity Results

The 45 subsamples from Tank V-9 were analyzed for U-234, U-235, and U-238 isotopic activity using alpha spectrometry; the results are given in Table G-2, along with the associated uncertainty in the activity measurement and the maximum, average, and standard deviations for the sample group (i.e., the five subsamples). Individual sample sizes, in grams, are provided in the results table in Attachment G-1 of this report. The samples from the upper zones in the tank (Zones 1, 3, and 6) that were primarily liquid (1RD001, 1RD003, and 1RD007) typically display lower uranium activities than the samples collected from the bottom zones that had more of a sludge-like appearance. For the three upper samples, uranium activity is lowest in the sample behind the baffle and highest opposite the baffle with the centerline sample falling in the middle. The ranges for the average and maximum uranium isotopic activities are as follows:

1. Average U-234 from 85.7 (Zone 3) to 465 pCi/g (Zone 6); maximum U-234 from 89.6 (Zone 3) to 508 pCi/g (Zone 6)

2. Average U-235 from 4.88 (Zone 3) to 25.1 pCi/g (Zone 6); maximum U-235 from 5.66 (Zone 3) to 28.3 pCi/g (Zone 6)

3. Average U-238 from 0.72 (Zone 3) to 8.90 pCi/g (Zone 6); maximum U-238 from 0.883 (Zone 3) to 10.7 pCi/g (Zone 6).

For the remaining six samples from Zones 2, 4, 5, 7, and 8, the average and maximum uranium isotopic activities are as follows:

1. Average U-234 from 3,430 (Zone 8) to 19,400 pCi/g (Zone 5); maximum U-234 from 3,980 (Zone 8) to 27,500 pCi/g (Zone 5)

2. Average U-235 from 121 (Zone 8) to 1,010 pCi/g (Zone 5); maximum U-235 from 139 (Zone 8) to 1,250 pCi/g (Zone 5)

3. Average U-238 from 12.7 (Zone 8) to 930 pCi/g (Zone 5); maximum U-238 from 14.8 (Zone 8) to 1,330 pCi/g (Zone 4).

These results mirror the trend found in the upper zones with respect to U-234 activity being the highest and U-238 activity the lowest. However, now the lowest uranium content is found in the zone opposite the baffle and the highest uranium content in the zones behind the baffle.

Validation of the isotopic data focused on detector system calibrations and operational performance checks, laboratory method blank results, laboratory generated duplicate results, laboratory analytical yields, and laboratory control sample (LCS) results. The only parameter that resulted in application of a data qualifier is the LCS result. In the case of U-235, one of the three LCS results exceeded the prescribed recovery limits of 70 to 130% at 137.7%. As a result, all U-235 results associated with the high LCS recovery batch (i.e:, Samples 1RD001, 1RD002, and 1RD003) are qualified as "J" indicating that these results may be biased high. Additionally, seven of the U-238 results are also qualified as "J" because the measured activities are only two to three times greater than their associated uncertainties. All other results are unqualified since they are more than three times the associated uncertainties.

G-4.3 TCLP Metal Results

The laboratory-prepared composite samples (1RD01001TI and 1RD01002TI) were analyzed for arsenic, barium, cadmium, chromium, lead, mercury, selenium, and silver. Both composites were subjected to sample extraction as specified by SW-846 Method 1311, sample preparation and analysis of mercury as specified by SW-846 Method 7470A, sample preparation for the remaining analytes as specified by SW-846 Method 3015A, and sample analysis for these analytes as specified by SW-846 6010A.

The results are presented in Table G-3. Arsenic, lead, and selenium from Sample 1RD01001TI and silver in both samples were not detected ("U" code in Table G-3), and the results shown are the method detection limits (MDLs). Arsenic, lead, and selenium from Sample 1RD01002TI were detected but at levels less than 10 times the MDL ("B" code in Table G-3). The remaining metal analytes (barium, cadmium, chromium, and mercury) were detected in both samples at levels greater than 10 times the MDL. Sample 1RD01002TI exceeds the regulatory limits for "characteristic" hazardous waste for both cadmium and mercury. All other results are below the hazardous waste thresholds for these metals. However, cadmium and mercury results from both composite samples exceed the universal treatment standards (110 and 25 µg/L, respectively) that may be applicable to the treatment and/or disposal of the tank contents.

Data validation qualifiers are attached to eight of the metal analytical results. The barium concentrations in both samples are qualified with a "J" flag in Table G-3 to denote that the analyte is detectable at the reported values but that the values are only estimates due to low recoveries for the continuous calibration verification, matrix spike, and matrix spike duplicate. Cadmium concentrations in both samples are qualified with a "J" flag to denote that the analyte is detectable at the reported values but that the values are only estimates due to low recoveries for the matrix spike, matrix spike duplicate, and analytical spike. The silver concentrations in both samples are qualified with a "UJ" flag to denote that the analyte is non-detectable at the reported values but that the reported values are only estimates due to low recoveries for the matrix spike, matrix spike duplicate, and analytical spike. Finally, the arsenic and selenium concentrations for Sample 1RD01002TI are qualified with a "U" flag to denote that the sample results are greater than the MDLs, but less than five times the amount of analyte found in the preparation blanks.

G-4.4 Additional Isotopic Uranium Analyses

In August 2001, BWXT Services, Inc., was requested to perform additional isotopic uranium analyses on the original Tank V-9 samples. For each of the original nine samples taken from Tank V-9, one of the five subsamples retained after the initial analysis was performed was randomly selected and fractions were removed for preparation by strong mineral acid dissolution and molten salt fusion. The resulting isotopic uranium analyses for both preparation methods are presented in Table G-4, along with the associated uncertainty and relative percent difference. A duplicate fusion analysis was also performed on the Zone 2 sample (0107074-04D).

The same general trends are observed in the additional isotopic analyses that were present in the original analyses. The uppermost sampling zones (Zones 1, 3, and 6) containing primarily liquid display lower uranium activities than the bottom zones containing primarily sludge, with the lowest activity in the zone behind the baffle (Zone 3) and the highest activity in the zone opposite the baffle (Zone 6). Maximum U-234 activities in the upper zones range from 74.8 (Zone 3) to 567 pCi/g (Zone 6); maximum U-235 activities from 4.95 (Zone 3) to 30.9 pCi/g (Zone 6); and maximum U-238 activities from 0.95 (Zone 3) to 13.3 pCi/g (Zone 6).

In the bottom sampling zones, the trend is reversed with the lowest uranium activity found in the zone opposite the baffle (Zone 8) and the highest activity in the zone behind the baffle (Zone 5). Maximum U-234 activities range from 3,490 (Zone 8) to 56,500 pCi/g (Zone 5); maximum U-235 activities from 119 (Zone 8) to 2,520 pCi/g (Zone 5); and maximum U-238 activities from 10.5 (Zone 8) to 1,060 pCi/g (Zone 5).

Generally for any given sample, the dissolution and fusion isotopic uranium activities are in fairly close agreement. The relative percent differences in uranium activity between the two preparation methods vary from 1.1 to 24.8% for U-234, from 1.5 to 25.0% for U-235, and from 1.5 to 57.2% for U-238. Finally, the relative percent difference between the duplicate Zone 2 fusion analyses (1RD05102R9 and 0107074-04D) varied from 7.2% for U-234 to 79.7% for U-238.

G-5. SAMPLING LESSONS LEARNED

The primary lesson learned during the sampling activity was that verbatim compliance with a written procedure might seem cumbersome when a difficulty is encountered, but timely execution of a change order to the procedure did not severely hamper the overall completion of the project. The approval process for changes to the procedures ensured that decisions were not made casually. ALARA and criticality concerns were given highest priority even when addressing a simple mechanical issue.

The video camera in the tank was most helpful. At the time the procedures were written, the reliability of the camera was underestimated. The only part of the procedure that required the camera was the initial reconnaissance and line-up of the glove bag with respect to the baffle. Beyond that, if the camera failed, the procedure could still be followed exactly; the equipment was designed to be operated without visual aid. Because the camera did survive in the radiation field, the images it provided gave an extra level of confidence to the sampling team. At each step, the visual confirmation reduced the need to proceed on faith.

The sludge encountered behind the baffle (Sampling Zones 3, 4 and 5) was substantially different from the training sludges used at the STAR Center. For future sampling activities, a wider range of practice material may better prepare the operators for unexpected conditions.

All of the sampling equipment was designed to fit into 55-gal drums for disposal. Some of the pieces, however, were packaged together in bundles that were too large to fit. Future equipment should use slightly smaller pieces to allow for packaging without exceeding the drum dimensions.

G-6. REFERENCES

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